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PREDICTION OF FAILURE TIMES
OF
ADHESIVE BONDS AT CONSTANT STRESS.
II. FAILURE AT LOW HUMIDITY



ELISE MCABEE
DAVID W. LEVI

DECEMBER 1970

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PREDICTION OF FAILURE TIMES OF
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FAILURE AT LOW HUMIDITY

by

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OBJECT

The object of this work was to examine possible methods of applying a reaction rate method to failure times of adhesive bonds at constant stress and low humidity.

SUMMARY

Two new procedures for estimating kinetic parameters from constant stress mechanical data are described. These procedures were applied to data on adhesive bonds obtained earlier. It was found that even for bonds tested at 20% humidity, consistent and reasonable results were obtained. By the previously used procedures, this correlation had been very doubtful.

INTRODUCTION

Tobolsky and Eyring (Ref 1) first applied reaction rate theory to polymer mechanical behavior. Such methods have recently been applied to describe the behavior of adhesive bonds under conditions of cohesive failure (Refs 2,3). The second report shows that a reaction rate method adequately predicts failure times at constant stress under conditions of 50% and 90-95% relative humidity. However, a correlation of the data taken at 20% relative humidity was planned but not undertaken because of an apparent uncertainty in the plots due to data scatter. Recently, this body of data has been reexamined and two additional treatments have been developed and applied. This report gives the results of this reexamination.

RESULTS AND DISCUSSIONS

An integrated form of the rate equation that has been used in previous studies may be written

$$\log t_f = C - \log T + \frac{\Delta H^\ddagger}{2.3RT} - b \frac{S}{T} \quad (1)$$

where t_f is failure time

C and b are constants

T is absolute temperature

$\Delta H^\ddagger/2.3RT$ is an activation energy term

S is the stress.

At constant temperature, the experimental data should give a straight line on plotting $\log t_f$ vs S/T as required by

$$\log t_f = D - bS/T \quad (2)$$

The apparent activation energy may then be evaluated by extrapolating several constant temperature lines to the vertical intercept ($S/T = 0$) and plotting according to

$$\log t_f T = \frac{\Delta H^\ddagger}{2.3RT} + C \quad (3)$$

Equations 2 and 3 were used to treat the data in an earlier report (Ref 3).

An alternative procedure was also used in the previous work (Ref 3). In this case, if we multiply Equation 1 through by T and consider a data point t_{f_1} and S_1 at T_1 , we obtain

$$T_1 \log t_{f_1} T_1 = CT_1 + \frac{\Delta H^\ddagger}{2.3R} - bS_1 \quad (4)$$

A similar expression may be written for t_{f_2} , S_2 , and T_2

$$T_2 \log t_{f_2} T_2 = CT_2 + \frac{\Delta H^\ddagger}{2.3R} - bS_2 \quad (5)$$

Assuming the constancy of H^\ddagger

$$\frac{\Delta H^\ddagger}{2.3R} = T_1 \log t_{f_1} T_1 - CT_1 + bS_1 = T_2 \log t_{f_2} T_2 - CT_2 + bS_2 \quad (6)$$

Rearranging and dividing through by $T_1 - T_2$

$$\frac{T_1}{T_1 - T_2} \log t_{f_1} T_1 - \frac{T_2}{T_1 - T_2} \log t_{f_2} T_2 = C + b \frac{(S_2 - S_1)}{(T_1 - T_2)} \quad (7)$$

For every possible pair of data points, the left hand side of equation 7 may be plotted against $(S_2 - S_1)/(T_1 - T_2)$.

C and b may then be evaluated as the intercept and slope, respectively. After C and b are determined, we may go back to Equation 1 in the form

$$\log t_f T - C + b \frac{S}{T} = \frac{\Delta H^\ddagger}{2.3RT} \quad (8)$$

The left hand side of Equation 8 is then plotted against $1/T$ to evaluate ΔH^\ddagger .

As had been indicated in previous work (Ref 3), an attempt to plot the data at 20% relative humidity according to Equation 7 gave a correlation coefficient for the least squares line of only 0.54. The use of Equations 2 and 3 gave the plots shown in Figures 1 and 2. The values of parameters obtained from these plots are reasonable but the scatter reduces confidence in the exact numerical values. Thus, the line in Figure 2 could be drawn a number of ways. It would appear that an additional treatment would be helpful.

A treatment using Equation 1, but holding $\log t_f T$ constant instead of temperature, has been used. In this case, we may rewrite Equation 1

$$\frac{S}{T} = \frac{C - \log t_f T}{b} + \frac{\Delta H^\ddagger}{2.3Rb} \cdot \frac{1}{T} \quad (9)$$

From plots of $\log t_f T$ versus S/T at various temperatures, we may determine values of S/T at various selected constant $\log t_f T$ values. Then we should get a straight line on plotting S/T versus $1/T$ for each constant $\log t_f T$. Obviously, the intercepts of these plots will be related to C , b and $\log t_f T$ as follows

$$\frac{C - \log t_f T}{b} = \text{Intercept} \quad (10)$$

or

$$\log t_f T = C - b \text{ Intercept} \quad (11)$$

From the linear plot of $\log t_f T$ versus Intercept, C and b may be determined. After b is known, it can be put back in the slope term of Equation 9 for the evaluation of ΔH^\ddagger .

A check on the use of Equations 9 and 11 was made by treating the data on AF126 adhesive reported previously for 90-95% and 50% relative humidity (Ref 3). The $\log t_f T$ versus S/T plots at 90-95% relative humidity are shown in Figure 3. These plots were used to obtain S/T at selected constant $\log t_f T$ values. The appropriate S/T versus $1/T$ lines are shown in Figure 4. These lines are all drawn with the same slope (10,700) as is required by Equation 9. Finally, b and C were evaluated from the slope and intercept of the straight line in Figure 5. Then from the slope of the S/T versus $1/T$ lines, ΔH^\ddagger was evaluated, thus:

$$\frac{\Delta H^\ddagger}{RTb} = \frac{\Delta H^\ddagger}{4.6(0.43)} = 10,700$$

$$\Delta H^\ddagger = 21 \text{ kcal}$$

This value of ΔH^\ddagger compares well with the 24 kcal reported earlier (Ref 3). b in this case is 0.43, whereas it was found to be 0.45 by the earlier methods. These values are considered in good agreement considering the scatter that is usually found in adhesive mechanical data. Values of C show a wider scatter. $C = -5.8$ in this work is to be compared with values of -7.4 and -8.1 found previously (Ref 3). It does appear that this method gives essentially the same results as the procedures formerly used.

The agreement of the method with the earlier work was further demonstrated using the data for AF126 adhesive at 50% relative humidity. Figures 6 through 8 illustrate the application. In this case, it was found that $b = 0.69$, $\Delta H^\ddagger = 51 \text{ kcal}$, and $C = -22.8$. The values reported in Reference 3 obtained by the other methods are $b = 0.71$, 0.71 ; $\Delta H^\ddagger = 51 \text{ kcal}$, 51 kcal , and $C = -22.5$, -22.1 . Once again the agreement is quite good.

Since the primary purpose of this work was to compare the 20% relative humidity data with the higher humidity values, the same treatment was applied to the 20% data. Figures 9 through 11 illustrate the data and method exactly as in the preceding cases. In this case, b is found to be 0.68, $\Delta H^\ddagger = 47 \text{ kcal}$, and $C = -20.5$. By Equations 2 and 3 (see Figures 1 and 2), the corresponding values are $b = 0.65$, $\Delta H^\ddagger = 46 \text{ kcal}$, and $C = -20.1$. This agreement gives much more confidence in the quantitative treatment of the 20% relative humidity data.

It is perhaps of interest to examine still another possible method of data treatment. If we hold the stress (S) constant, we may rewrite Equation 1 in the form

$$\log t_f T = C + \left(\frac{\Delta H^\ddagger}{2.3R} - bS \right) \frac{1}{T} \quad (12)$$

On plotting $\log t_f T$ vs $1/T$, we should get

$$\text{Slope} = \frac{\Delta H^\ddagger}{2.3R} - bS \quad (13)$$

And, on plotting slope vs S, we could evaluate ΔH^\ddagger and b. Of course, C would be obtained directly from the intercept of Equation 12.

Unfortunately, the above procedure is difficult to use in cases such as constant stress experiments with AF126 adhesive described above because of the difficulty in distinguishing the small changes in slope in Equation 12. For example, in the cases under consideration, this change would be of the order of 10% or less. Such a change might be almost completely obscured by the experimental scatter. It would appear that a better approach is to multiply Equation 12 through by T

$$T \log t_f T = \left(\frac{\Delta H^\ddagger}{2.3R} - bS \right) + CT \quad (14)$$

Now on plotting $T \log t_f T$ vs T, we get

$$\text{Intercept} = \frac{\Delta H^\ddagger}{2.3R} - bS \quad (15)$$

C is obtained from the slope in Equation 14. ΔH^\ddagger and b are obtained from the straight line relation between the intercepts of Equation 14 and S, in accord with Equation 15.

The lines, according to Equation 14 at various constant S values are shown in Figure 12. The graph for evaluation of ΔH^\ddagger and b (Equation 15) is given in Figure 13. Values of the parameters are $b = 0.61$, $C = -19.8$, and $\Delta H^\ddagger = 45$ kcal. These values are in reasonable agreement with those given earlier in this report.

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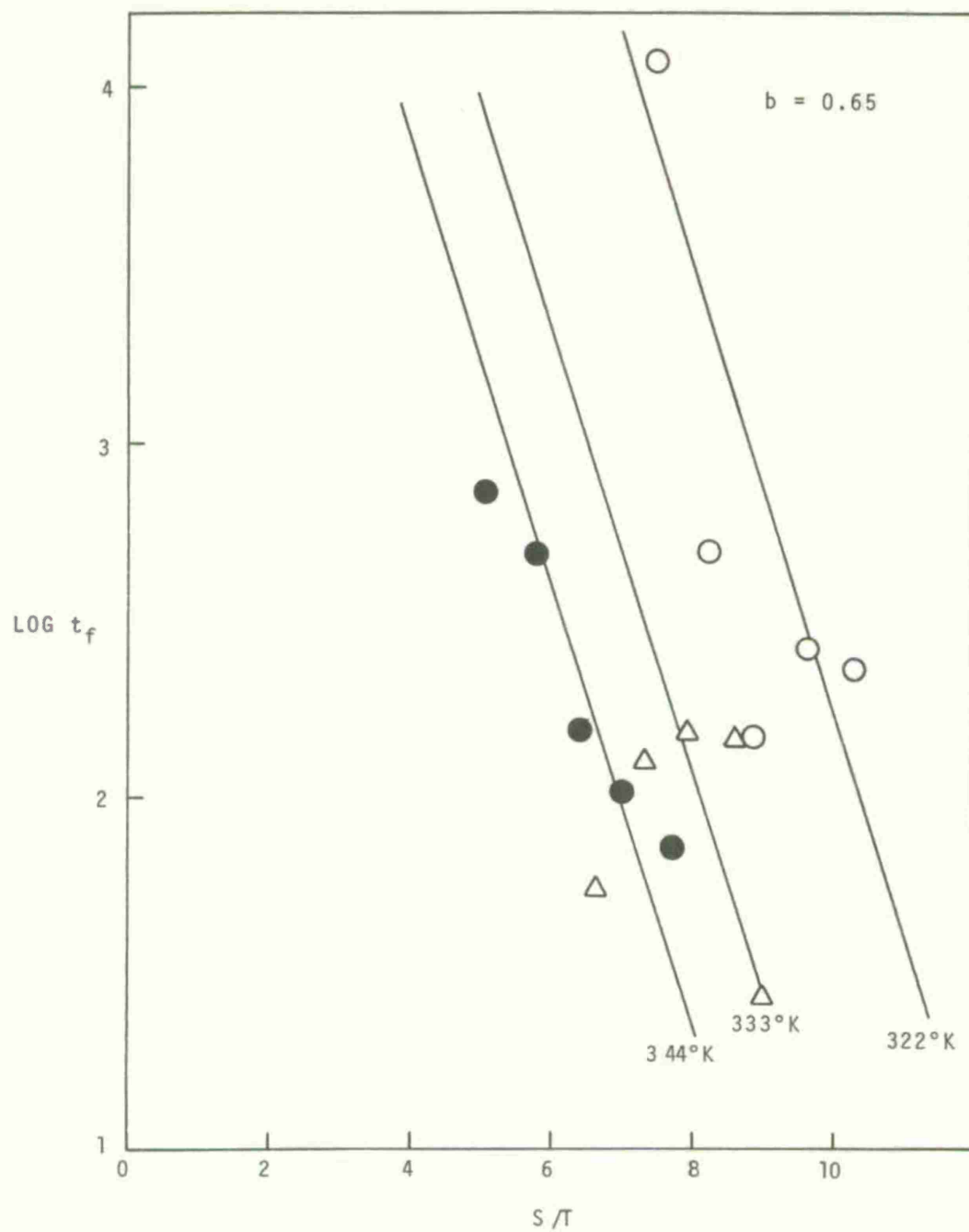


Fig 1 $\text{Log } t_f$ vs S/T for AF 126 adhesive (aluminum adherends) under constant stress at 20% relative humidity

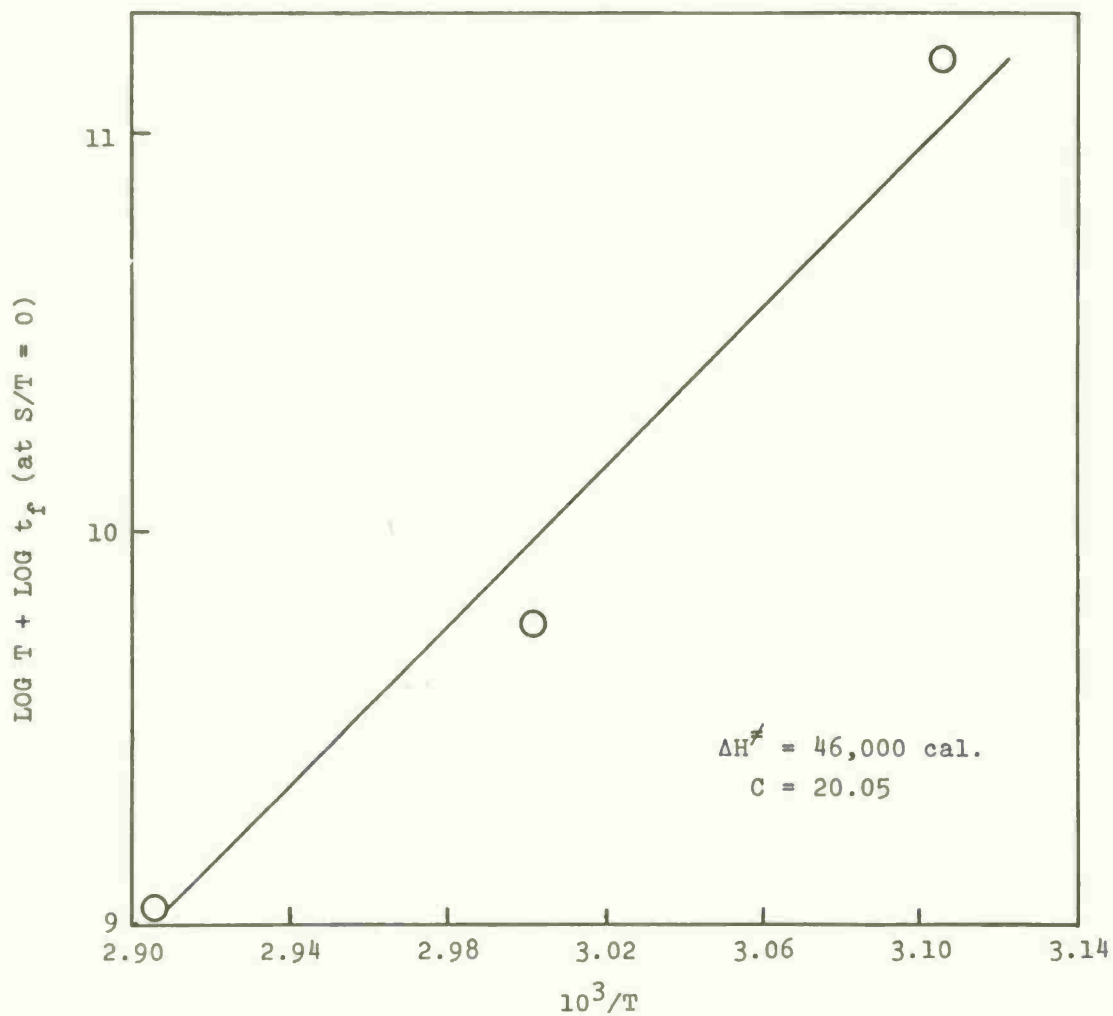


Fig 2 Arrhenius type plot for evaluation of ΔH^\ddagger at 20% relative humidity

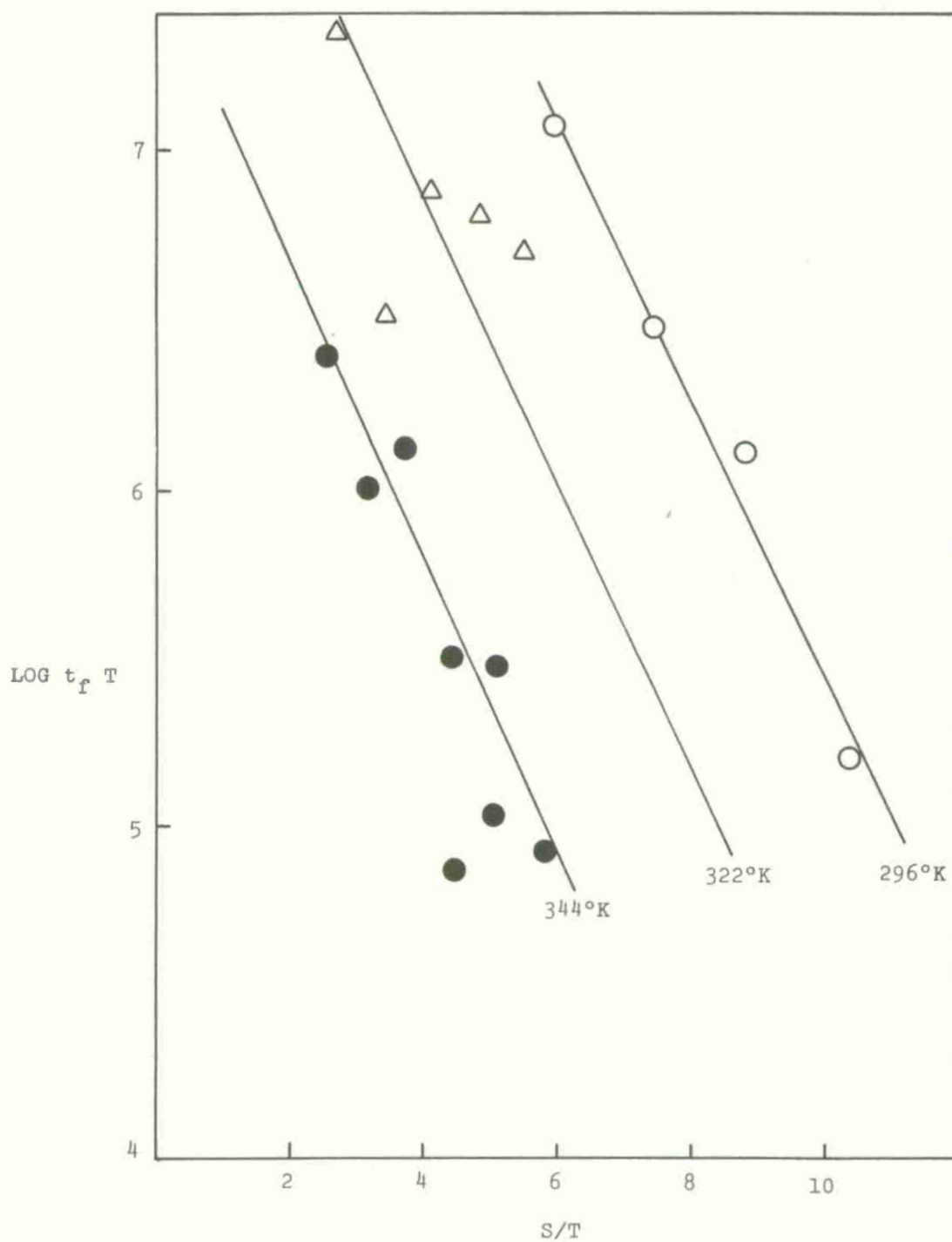


Fig 3 Log $t_f T$ vs S/T for AF126 adhesive (aluminum adherends)
at 90 - 95% relative humidity

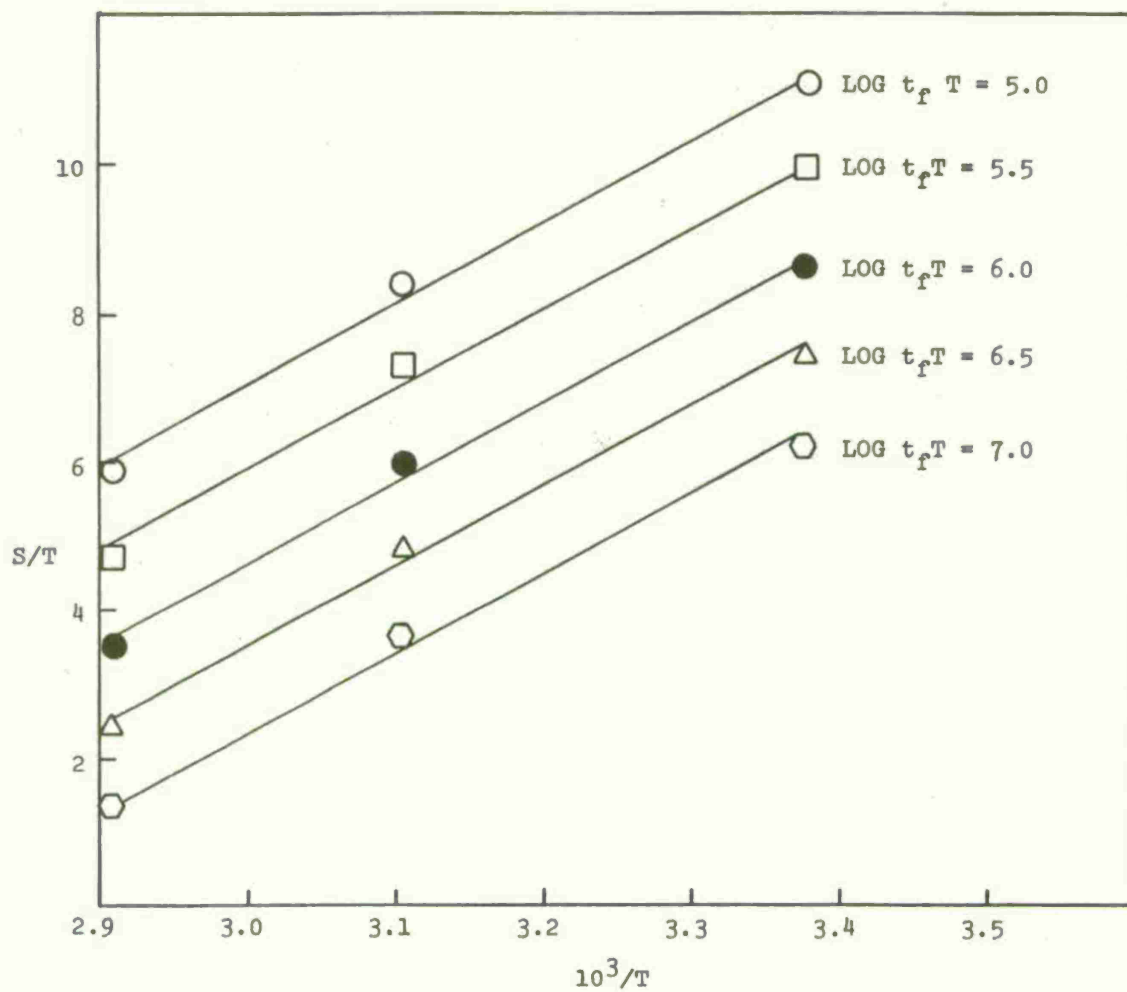


Fig 4 S/T vs $1/T$ for AF126 adhesive at 90 - 95% relative humidity

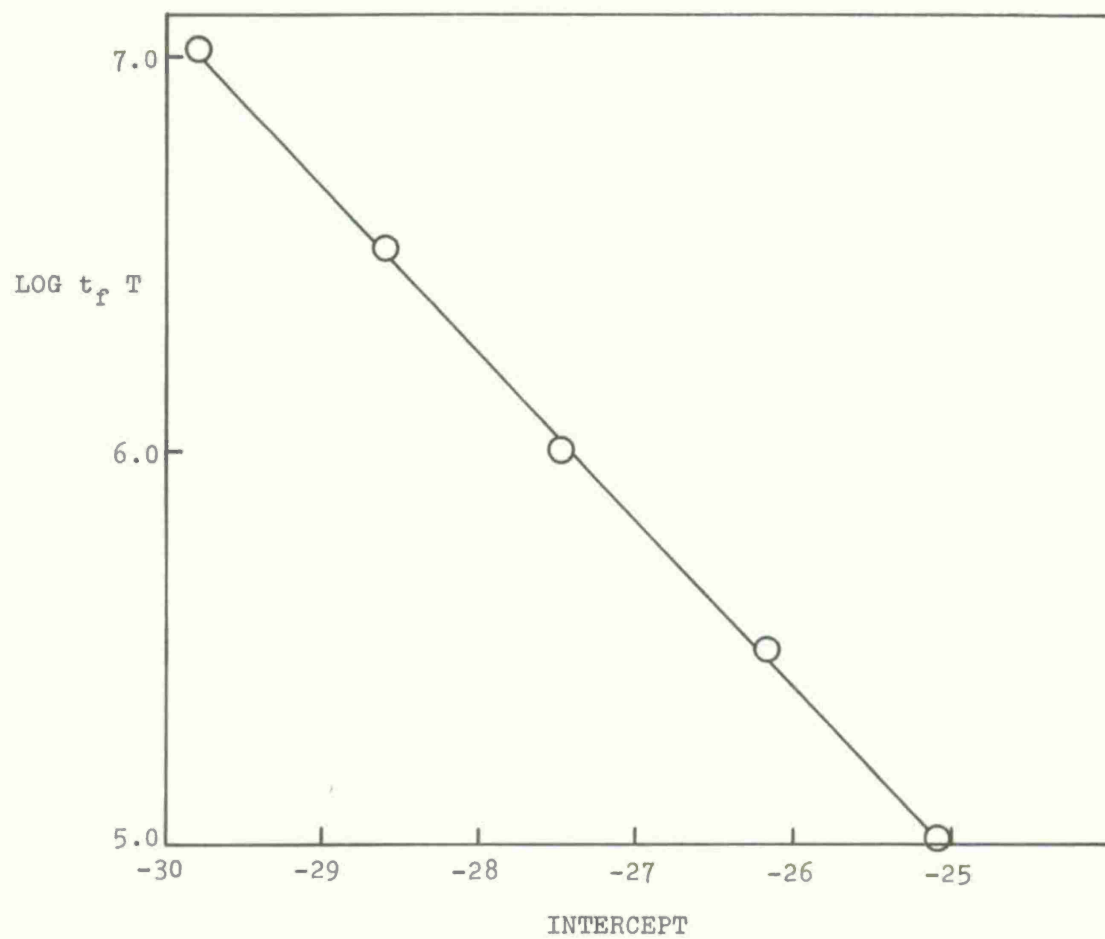


Fig 5 $\text{Log } t_f T$ vs intercept for AF126 adhesive at 90 - 95% relative humidity

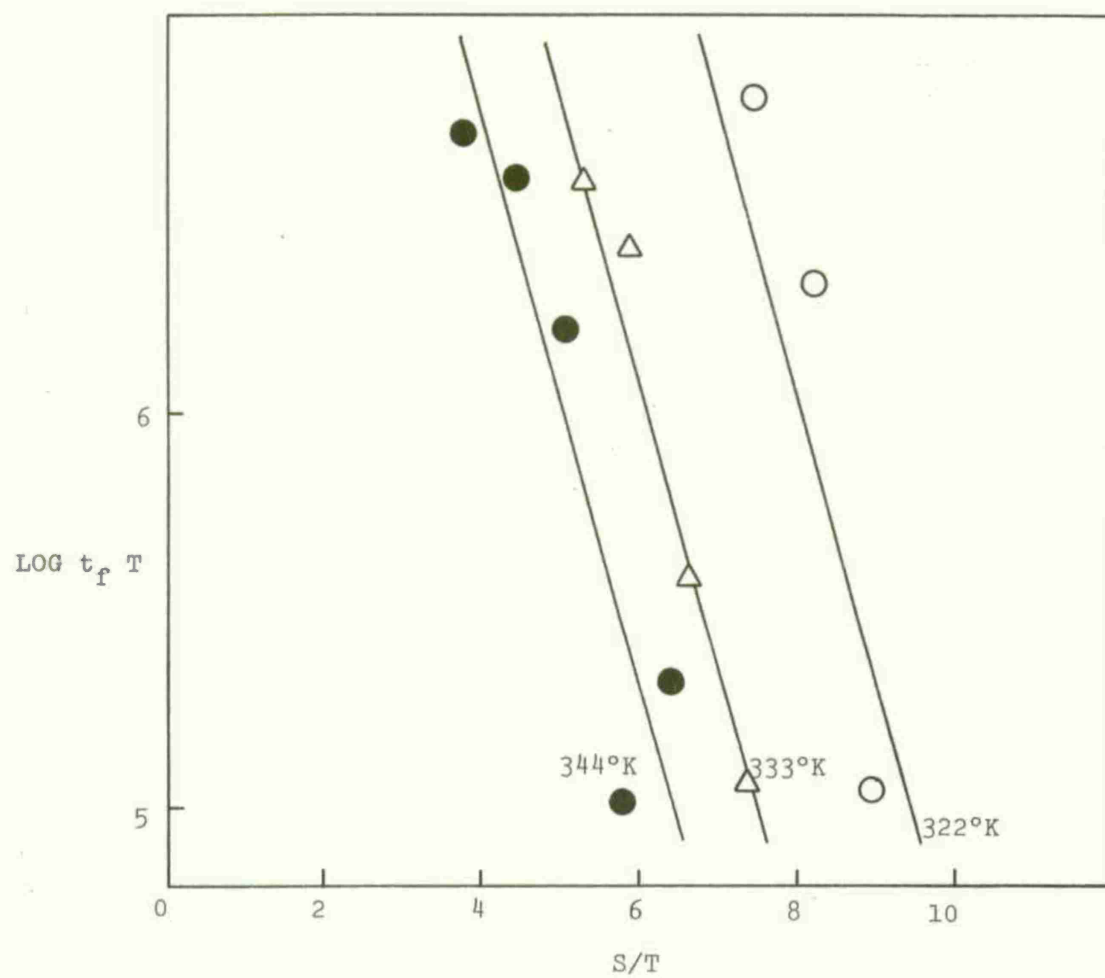


Fig 6 Log $t_f T$ vs S/T for AF126 adhesive at 50% relative humidity

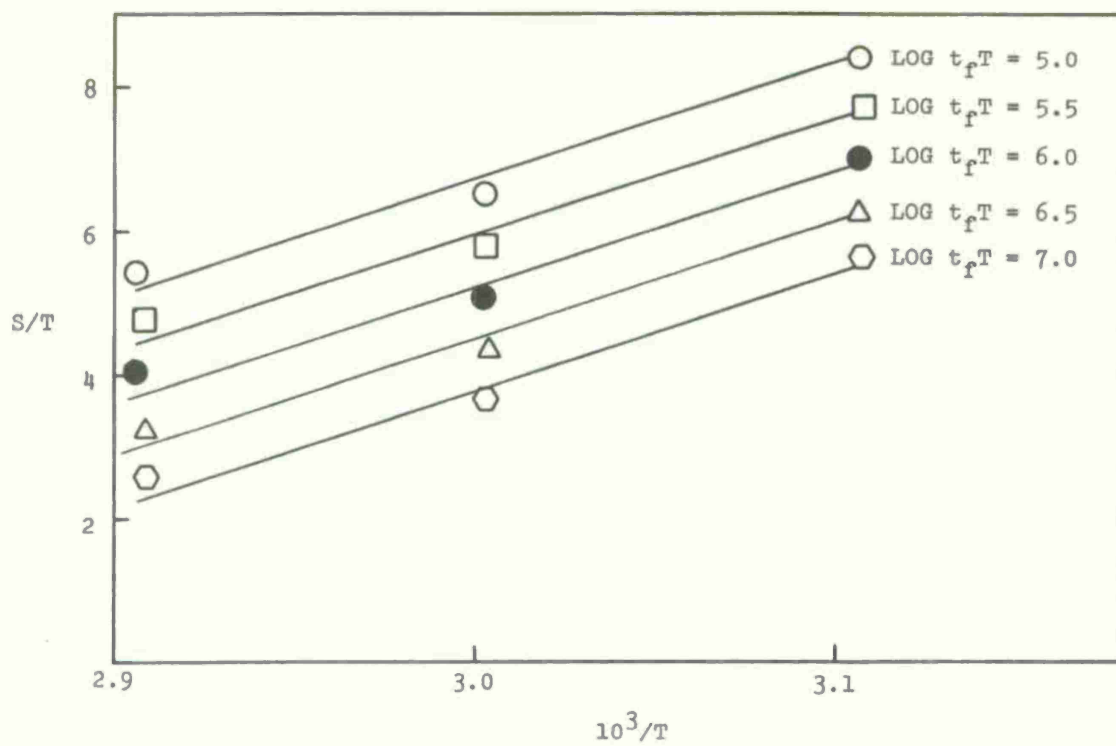


Fig 7 S/T vs $1/T$ for AF126 adhesive at 50% relative humidity

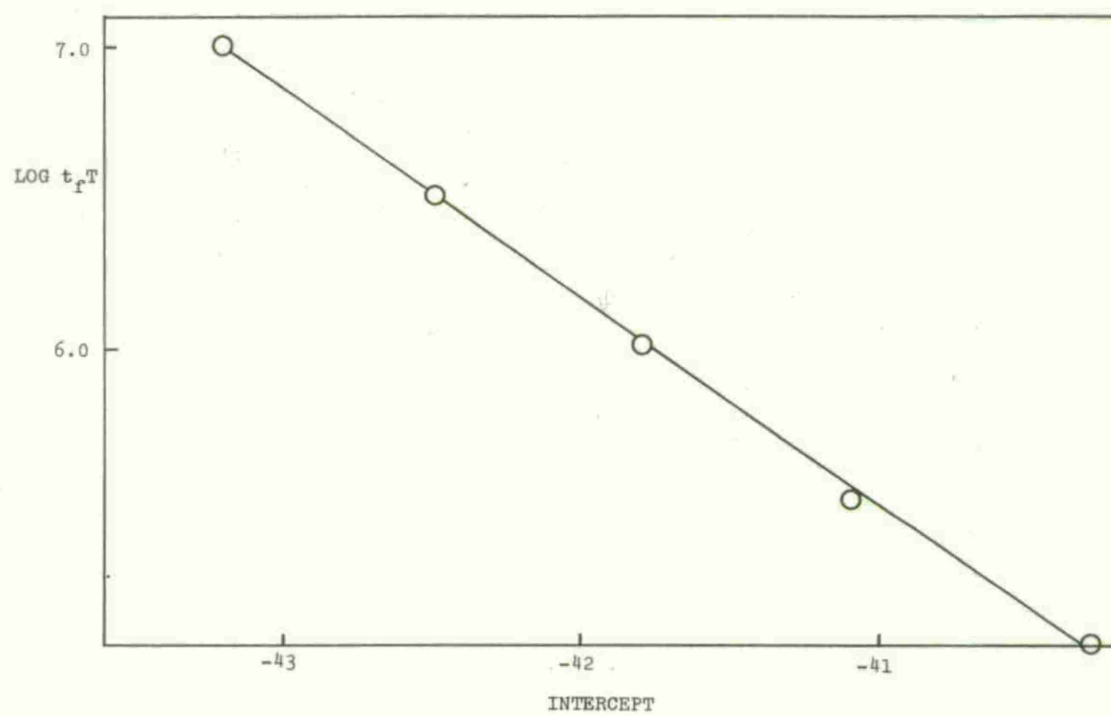


Fig 8 $\text{Log } t_f T$ vs intercept for AF126 adhesive at 50% relative humidity

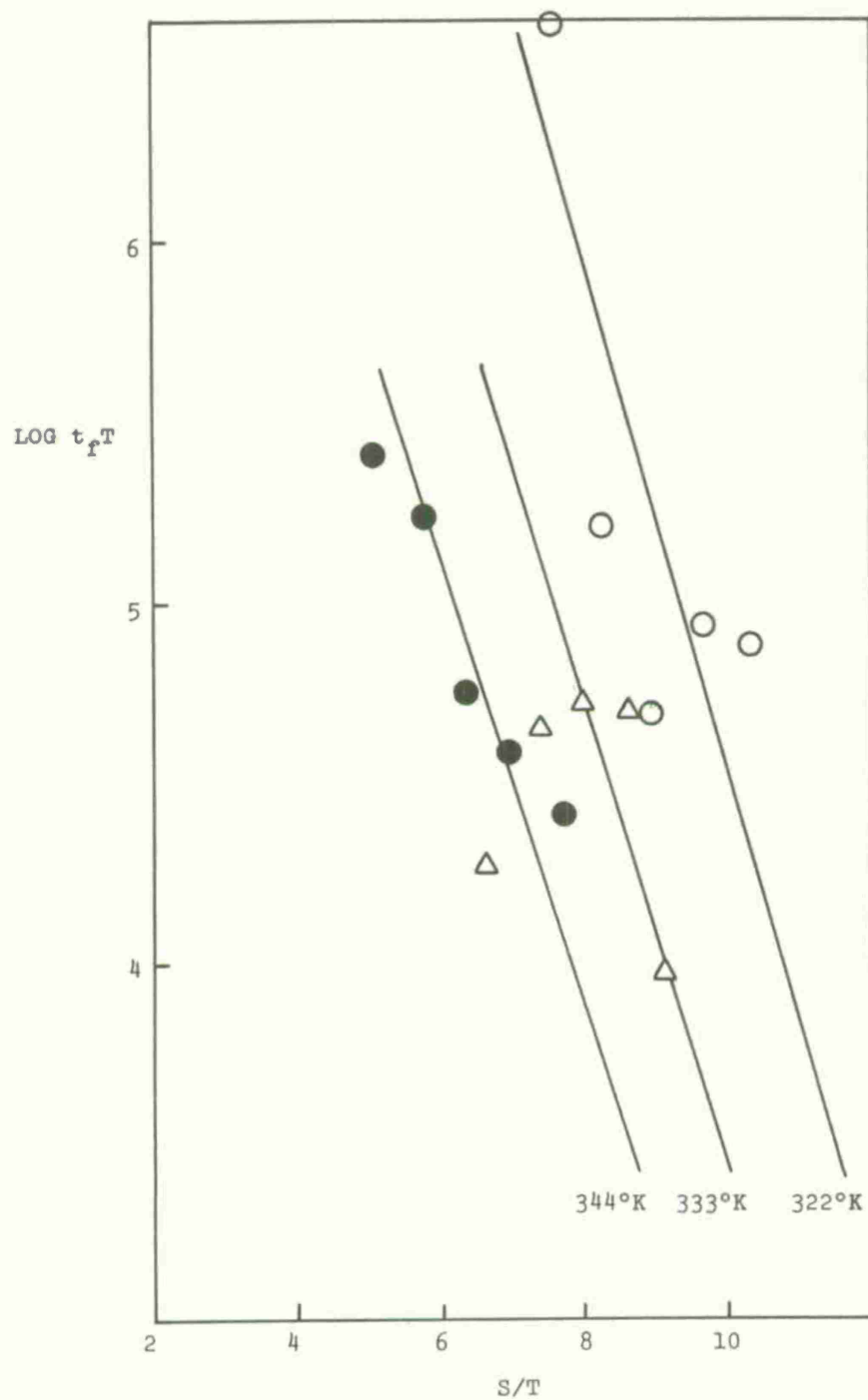


Fig 9 Log $t_f T$ vs S/T for AF126 adhesive at 20% relative humidity

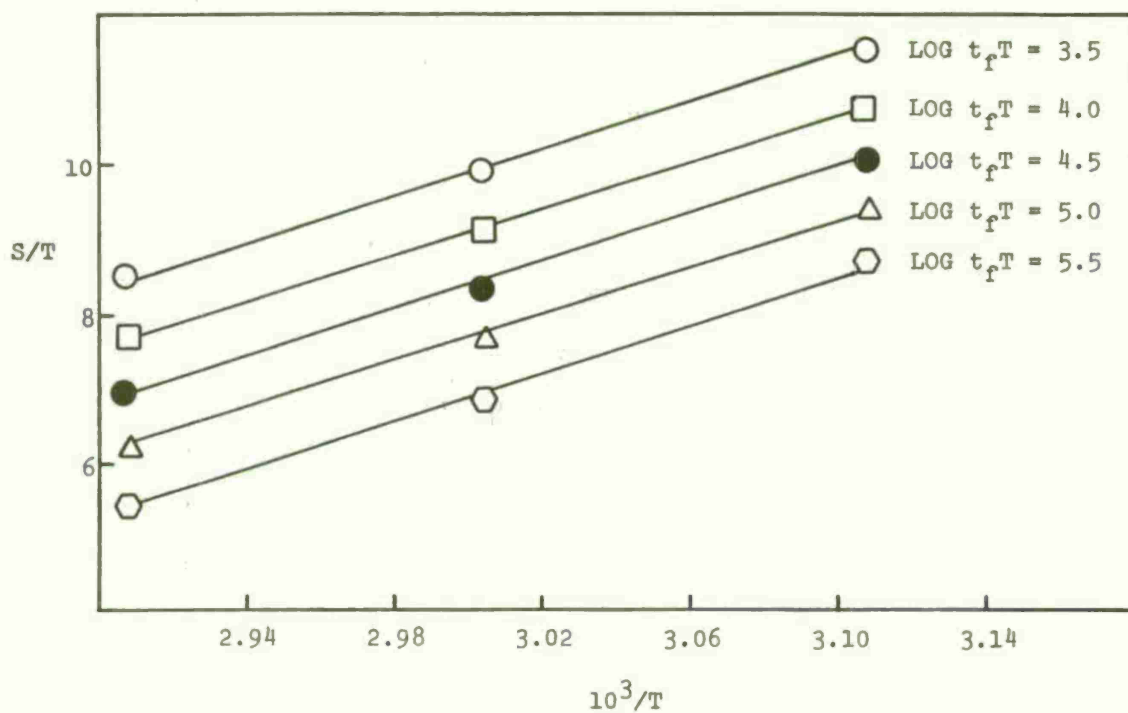


Fig 10 S/T vs 1/T for AF126 adhesive at 20% relative humidity

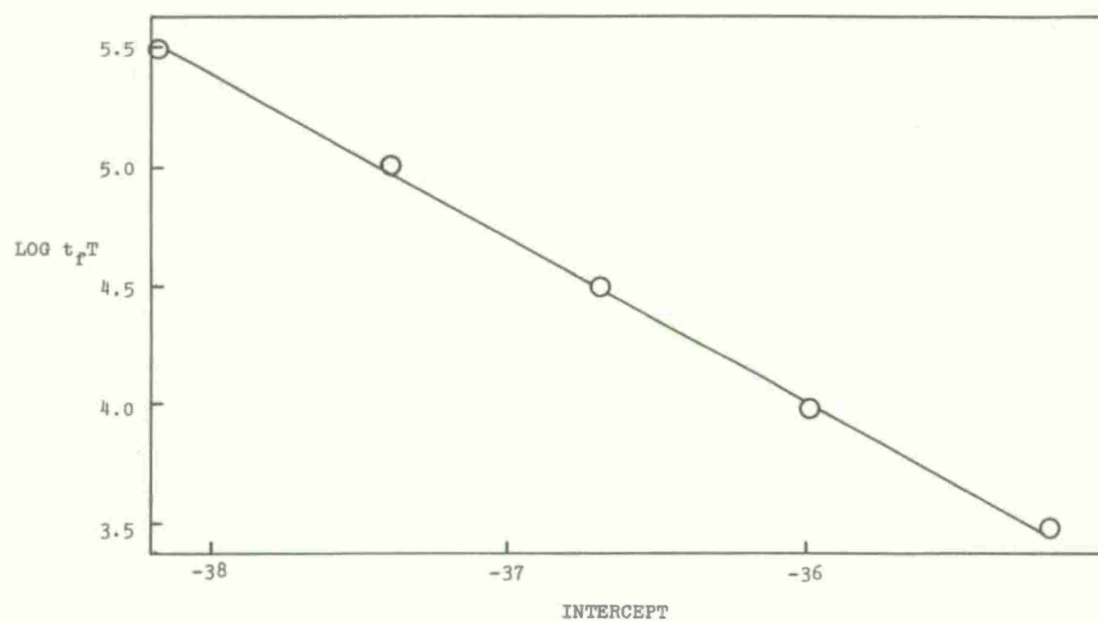


Fig 11 $\text{Log } t_f T$ vs intercept for AF126 adhesive at 20% relative humidity

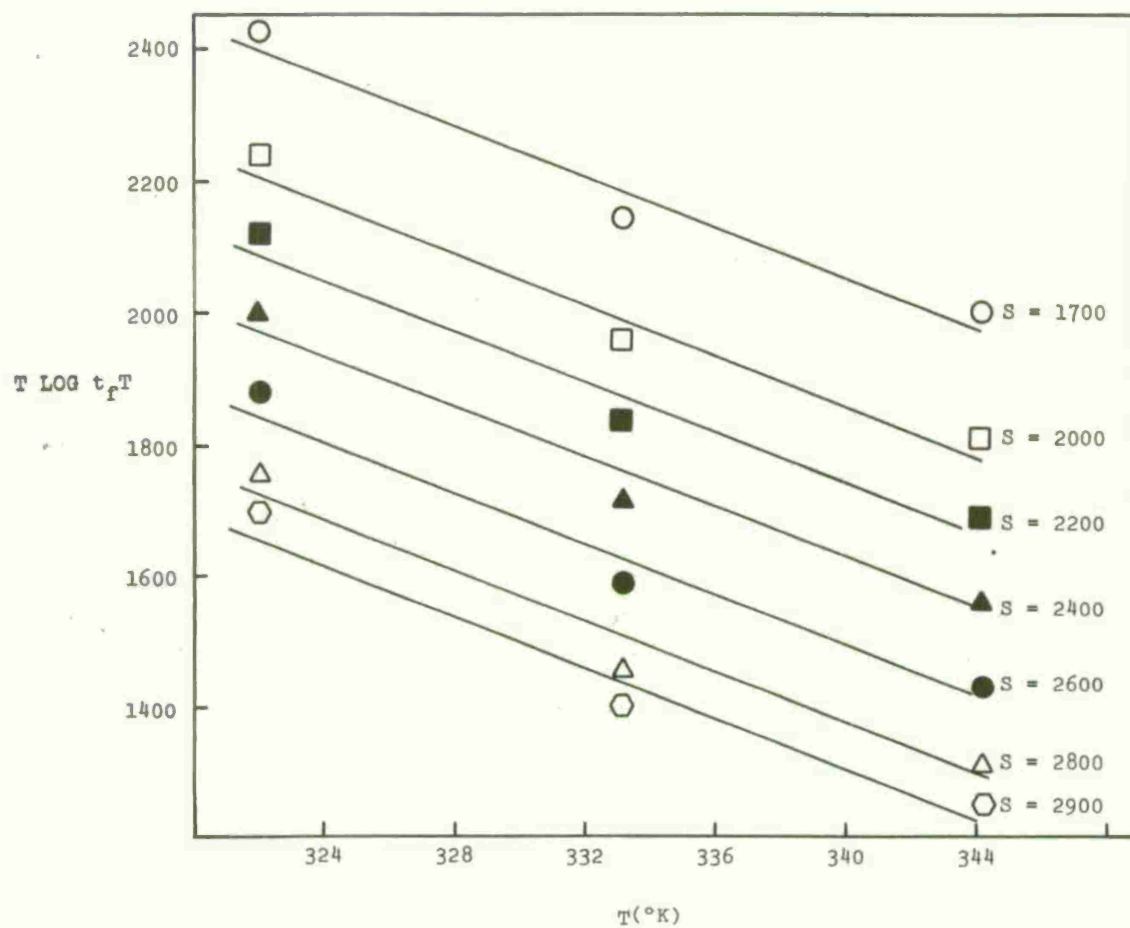


Fig 12 $T \log t_f T$ vs T for AF126 adhesive at 20% relative humidity

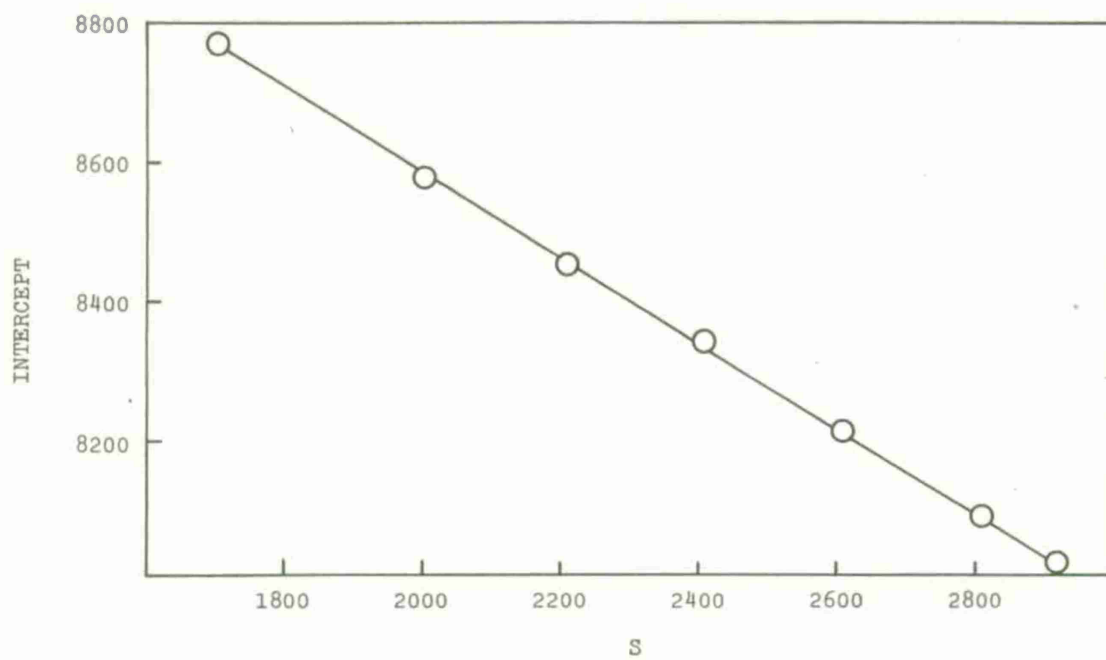


Fig 13 Intercept vs S for AF126 adhesive at 20% relative humidity

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13. ABSTRACT			
Two new procedures for estimating kinetic parameters from constant stress mechanical data are described. These procedures were applied to data on adhesive bonds obtained earlier. It was found that even for bonds tested at 20% humidity, consistent and reasonable results were obtained. By the previously used procedures, this correlation had been very doubtful.			

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